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DESIGNATED/ELECTED OFFICE (DO/EO/US)
CONCERNING A FILING UNDER 35 U.S.C. 371

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U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

10/049539

INTERNATIONAL APPLICATION NO.
PCT/IP00/02874INTERNATIONAL FILING DATE
May 1, 2000PRIORITY DATE CLAIMED
May 7, 1999

TITLE OF INVENTION

HOT PLATE AND CONDUCTIVE PASTE

APPLICANT(S) FOR DO/EO/US
YANLING ZHOU

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
 - a. ☒ is attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ has been communicated by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
 - a. ☒ is attached hereto.
 - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
 - a. ☒ are attached hereto (required only if not communicated by the International Bureau).
 - b. ☐ have been communicated by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☒ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A copy of the International Search Report (PCT/ISA/210).

Items 13 to 20 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
20. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
21. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
22. ☒ Certificate of Mailing by Express Mail
23. ☐ Other items or information:

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 1.492) 10/049539		INTERNATIONAL APPLICATION NO. PCT/JP00/02874		ATTORNEY'S DOCKET NUMBER ONDAT-017US	
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24. The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) : <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO \$1040.00 <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO \$890.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$740.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$710.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00				CALCULATIONS PTO USE ONLY	
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Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30				\$0.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	11 - 20 =	0	x \$18.00	\$0.00	
Independent claims	1 - 3 =	0	x \$84.00	\$0.00	
Multiple Dependent Claims (check if applicable) <input type="checkbox"/>				\$0.00	
TOTAL OF ABOVE CALCULATIONS =				\$890.00	
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$0.00	
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Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30 +				\$0.00	
TOTAL NATIONAL FEE =				\$890.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input checked="" type="checkbox"/>				\$40.00	
TOTAL FEES ENCLOSED =				\$930.00	
				Amount to be: refunded \$ charged \$	

a. ☒ A check in the amount of \$930.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

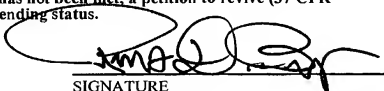
c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 50-0878. A duplicate copy of this sheet is enclosed.

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NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

Robert D. Buyan
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 SIGNATURE
Robert D. Buyan
 NAME
32,460
 REGISTRATION NUMBER
November 7, 2001
 DATE

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)	
Yanling Zhou)	Art Unit: Unassigned
)	
Application No.: Unassigned)	Examiner: Unassigned
Corresponding to PCT/JP00/02874)	
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Filed: Herewith, November 7, 2001)	
)	
For: HOT PLATE AND)	
<u>CONDUCTIVE PASTE</u>)	

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Commissioner for Patents
Washington, D.C. 20231

Preliminary Amendment

Dear Sir:

Prior to calculation of the filing fee and examination on the merits, please amend the above-identified patent application, without prejudice, as follows:

In the Specification:

- Page 1, line 1, change "SPECIFICATION" to --TITLE OF THE INVENTION--;
- line 5, change "TECHNICAL FIELD" to --BACKGROUND OF THE INVENTION--;
- line 10, delete "BACKGROUND ART";
- Page 3, line 4, change "DISCLOSURE OF THE INVENTION", to --BRIEF SUMMARY OF THE INVENTION--;
- Page 5, line 12, change "BEST MODE FOR CARRYING OUT THE INVENTION" to --DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS--;
- Page 12, line 10, after "butyl Carbitol." insert the sentence --Butyl Carbitol is a trademark of Union Carbide. It is also known as Diethylene Glycol Monobutyl Ether.--.

In the claims:

- Please amend Claims 2, 3, 4, 5 and 7, as shown herebelow and in the marked-up versions provided in Appendix A.
- 2. The hot plate according to claim 1, characterized in that the conductive layer further includes bismuth or its oxide.
- 3. The hot plate according to claim 1, characterized in that the ceramic substrate is a ceramic substrate is a ceramic nitride substrate or a ceramic carbide base plate.
- 4. The hot plate according to claim 1, wherein the glass frit includes zinc boron silicate.
- 5. The hot plate according to claim 1, wherein the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains.
- Please add new Claims 8-11 as shown herebelow and in the accompanying Appendix A.
- 8. A hot plate that uses a ceramic substrate provided with a conductive layer, wherein the hot plate characterized in that the conductive layer includes ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains, wherein the ceramic substrate is a ceramic nitride substrate or a ceramic carbide base plate..
- 9. The hot plate according to claim 8 , wherein the glass frit includes zinc boron silicate.
- 10. The hotplate according to claim 8, wherein the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains.

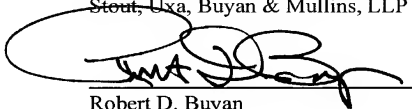
11. The hot plate according to claim 10, wherein the glass frit includes zinc boron silicate.

REMARKS

The claims have been rewritten to eliminate multiple claim dependency and to improve readability. The specification has been amended to include US-style headings, and to identify butyl Carbitol as a trademark of Union Carbide.

The Examiner is invited to telephone the undersigned attorney regarding any questions and to discuss steps necessary for placing the application in condition for allowance.

Respectfully submitted,
Stout, Uxa, Buyan & Mullins, LLP

A handwritten signature in black ink, appearing to read "Robert D. Buyan", is written over a horizontal line.

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Date: November 7, 2001

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Appendix A

2. (Amended) The hot plate according to claim 1, characterized in that the conductive layer further includes [ruthenium oxide,] bismuth or its oxide [, glass frit, and noble metal grains].
3. (Amended) The hot plate according to claim 1 [or 2], characterized in that the ceramic substrate is a ceramic substrate is a ceramic nitride substrate or a ceramic carbide base plate.
4. (Amended) The hot plate according to [any one of claims 1 to 3] claim 1, wherein the glass frit includes zinc boron silicate.
5. (Amended) The hot plate according to [any one of claims 1 to 4] claim 1, wherein the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains.
8. (New) A hot plate that uses a ceramic substrate provided with a conductive layer, wherein the hot plate characterized in that the conductive layer includes ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains, wherein the ceramic substrate is a ceramic nitride substrate or a ceramic carbide base plate..
9. (New) The hot plate according to claim 8 , wherein the glass frit includes zinc boron silicate.
10. (New) The hotplate according to claim 8, wherein the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains.
11. (New) The hot plate according to claim 10, wherein the glass frit includes zinc boron silicate.

2/p_{rt}s

SPECIFICATION

HOT PLATE AND CONDUCTIVE PASTE

5 TECHNICAL FIELD

The present invention relates to a hot plate, which uses a ceramic substrate, and a conductive paste.

10 BACKGROUND ART

During a semiconductor fabrication process, for example, when heating and drying a silicon wafer subsequent to the application of a photosensitive resin, a heating apparatus, which is referred to as a hot plate, is normally used.

In recent years, substrates made of ceramic, such as alumina, is often used to form a hot plate. A resistor, which functions as a conductive layer and which has a predetermined pattern, is formed on one side of the alumina substrate. A terminal connection pad is formed on part of the resistor. Such conductive layer is formed by applying, heating, and bonding an alumina substrate silver paste to the substrate. Subsequently, a terminal pin is soldered to the pad, and the terminal pin is connected to a power source by a wire. A silicon wafer, which is a heated subject, is placed on an upper surface of the hot plate. When the resistor is energized in this state, the silicon wafer is heated to 100°C or higher.

A conductive paste including 60wt% to 80wt% of silver, 1wt% to 10wt% of glass frit, the base of which is lead boron

silicate, 1wt% to 10wt% of a binder, and 10wt% to 30wt% of a solvent is often used to form a conductive pattern layer (refer to Japanese Unexamined Patent Publication No. 4-300249). Glass frit, which is a secondary component, is especially required to obtain the optimal adhesion for the conductive pattern layer.

When applying the above conventional lead paste directly to a ceramic substrate, such as an aluminum nitride substrate or a silicon carbide substrate, the following shortcomings occur. The heat produced when bonding the paste causes the oxides in the paste to react with aluminum nitride and produce a large amount of alumina and nitrogen gas. This is considered to occur mainly because of the large amount of oxides, especially lead oxide, in the glass frit. In this case, the high pressure of the nitrogen gas produced during the bonding of the paste forces the nitrogen gas to pass through the grain boundary of the silver grains toward the exterior of the conductive pattern layer. As a result, the conductive pattern layer is apt to expand and the accuracy for forming the pattern decreases.

Further, depending on the usage of the hot plate, there may be cases in which it is desired that the specific resistance of the conductive pattern layer be increased. In such case, if the amount of silver grains in the paste is relatively decreased to relatively increase the amount of glass frit, the ratio of the conductive components occupying the conductive pattern layer would decrease and, as a result, increase the specific resistance.

Simply employing this method would, however, most likely increase the amount of nitrogen gas produced when

bonding the paste and cause the conductive pattern layer to expand.

DISCLOSURE OF THE INVENTION

It is an object of the present invention to provide a hot plate having a conductive pattern layer that expands little, has superior adhesion, and has a large specific resistance and to provide a conductive paste optimal for the manufacturing of such hot plate.

A first perspective of the present invention is a hot plate that uses a ceramic substrate provided with a conductive layer. The conductive layer includes ruthenium oxide, glass frit, and noble metal grains.

The conductive layer includes ruthenium oxide. Thus, reaction between the glass frit and the ceramic substrate is suppressed even if the added amount of the glass frit is relatively large (10wt% or greater relative to the noble metal) to increase the specific resistance. This prevents expansion of the conductive layer. Accordingly, the conductive layer has a specific resistance that is $10 \mu \Omega \cdot \text{cm}$ or greater. Further, it is considered that the ruthenium oxide itself functions to increase the specific resistance.

It is preferred that the ceramic substrate includes ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains. By adding bismuth or its oxide, reaction between the glass frit and the ceramic substrate is suppressed. Thus, the conductive layer has superior adhesion.

It is preferred that the ceramic substrate be a

ceramic nitride substrate or a ceramic carbide substrate. The employment of a ceramic nitride substrate or a ceramic carbide substrate improves thermal conductivity and high-temperature heat resistance. The employment of the aluminum nitride substrate, which especially has superior heat resistance and high thermal conductivity, enables the hot plate to withstand usage under high temperatures and the hot plate is thus practical.

It is preferred that the glass frit includes zinc boron silicate. Differing from the conventional product, which includes lead boron silicate, the glass frit, which includes zinc boron silicate, reacts with the nitride in the ceramic substrate and has a small amount of oxide that produces nitrogen gas. Accordingly, even if the conductive layer is formed using a material including zinc boron silicate, a large amount of nitrogen gas is not produced and the expansion of the conductive layer is suppressed.

It is preferred that the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains. Since the used metal grains resists oxidization even when exposed to high temperatures and has a sufficiently large resistance, a conductive layer optimal for functioning as a resistor that produces heat is easily formed.

A second perspective according to the present invention is a conductive paste including ruthenium oxide, glass frit, and noble metal grains.

A third perspective according to the present invention is a conductive paste including ruthenium oxide, bismuth or

its oxide, glass frit, and noble metal grains.

BRIEF DESCRIPTION OF THE DRAWINGS

5 Fig. 1 is a schematic cross-sectional view showing a hot plate unit according to one embodiment of the present invention.

10 Fig. 2 is a partial enlarged cross-sectional view showing the hot plate unit of Fig. 2.

BEST MODE FOR CARRYING OUT THE INVENTION

15 A hot plate unit 1 according to one embodiment of the present invention will now be described with reference to Figs. 1 and 2.

20 The hot plate unit 1, which is shown in Fig. 1, is formed by the main elements of a casing 2 and a hot plate 3.

25 The casing 2 is a cup-like metal member having an opened portion 4, the cross-section of which is round, located at its upper portion. The casing 2 does not have to be cup-like and may have an opened bottom. A hot plate 3 is attached to the opening 4 by means of an annular seal ring 14. A lead wire hole 7 for receiving current supplying lead wires 6 extends through the peripheral part of the bottom portion 2a of the casing 2.

30 The hot plate 3 of the present embodiment, which is formed from a ceramic substrate 9, is used to dry a silicon wafer W1, to which a photosensitive resin is applied, at 50°C to 800°C or to perform heating during sputtering.

It is preferred that a ceramic nitride substrate or a ceramic carbide substrate be selected as the ceramic substrate 9 since they have superior heat resistance and high thermal conductivity properties. More specifically, it is preferred that an aluminum nitride substrate, a silicon nitride substrate, a boron nitride substrate, a titanium nitride substrate, a silicon carbide substrate, a boron carbide substrate, or a titanium carbide substrate be selected. Among these substrates, it is most preferred that the aluminum nitride substrate be selected and next preferred that the silicon nitride substrate be selected. This is because these substrates have the highest thermal conductivity among the above ceramic nitrides.

The ceramic substrate 9 is disk-like, has a thickness of about 1mm to 100mm, and has a diameter that is slightly smaller than the outer dimension of the casing 2.

Referring to Figs. 1 and 2, a wiring resistor 10, which serves as a conductive pattern layer, is formed in a concentric or spiral manner on the lower surface of the plate-like substrate 9. Pads 10a are formed on an end of the wiring resistor 10. The wiring resistor 10 and the pads 10a are formed by printing, heating, and bonding a conductive paste (noble metal paste) P1 on the surface of the ceramic substrate 9. In the hot plate 3 of the present embodiment, the surface for heating the silicon wafer W1 is located on the opposite side of the conductive pattern layer formation layer, or on the upper surface. Such structure has an advantage in that a difference in temperature between locations does not occur in the hot plate 3 and in that the silicon wafer W1 is uniformly heated.

The wiring resistor 10 and the pads 10a of the present embodiment that are formed from the noble metal paste P1 includes noble metal grains as a main component and glass frit, or the like, as a secondary component. It is preferred that the noble metal grains used in the present embodiment have an average grain diameter of $6\mu\text{m}$ or less and be flake-like.

It is preferred that the flake-like noble metal grains be selected from one of gold grains (Au grains), silver grains (Ag grains), platinum grains (Pt grains), and palladium grains (Pd grains). These noble metals relatively resist oxidation even if they are exposed to high temperatures and have a sufficiently large resistance when energized and heated.

Referring to Figs. 1 and 2, the basal end of a terminal pin 12, which is made of a conductive material, is soldered to each pad 10a. This electrically connects each terminal pin 12 to the wiring resistor 10. Sockets 6a, which are located on the distal end of the lead wires 6, are fit into the distal ends of the terminal pins 12. Accordingly, the temperature of the wiring resistor 10 increases and heats the entire hot plate 3 when current is supplied to the wiring resistor 10 via the lead wires 6 and the terminal pins 12.

An example of the procedures for manufacturing the hot plate 3 will now be briefly described.

A sintering-aid agent, such as yttria, and a binder are added as required to ceramic grains to prepare a mixture.

The mixture is uniformly kneaded into, for example, three rolls. The kneaded material is used to press mold plate-like molding products having a thickness of 1 to 100mm.

5 Holes are punched or drilled in the molded product to form pin insertion holes, which are not shown in the drawings. After the hole forming process, the molded product is dried. Then, the molded product undergoes provisional baking and main baking so that it is completely sintered.
10 This forms the ceramic sinter substrate 9. It is preferred that the baking process be performed in a hot-press apparatus and that the baking process be performed at a temperature of about 1500°C to 2000°C. Afterward, the ceramic substrate 9 is cut into a disk-like shape having a
15 predetermined diameter (in the present embodiment, 230mm ϕ) and undergoes surface grinding, preferably with a hub grinder.

20 After the above process, the noble metal paste P1, which has been prepared beforehand, is uniformly applied to the lower surface of the ceramic substrate 9, preferably through screen-printing.

25 In addition to noble metal grains, the noble metal paste P1 used here includes ruthenium oxide, glass frit, a resin binder, and a solvent. The noble metal paste P1 may also include bismuth or bismuth oxide.

30 It is preferred that 40wt% to 60wt% of noble metal grains and 10wt% to 30wt% (however, 10wt% or greater with respect to noble metal) of glass frit be included in the noble metal paste P1.

If the amount of noble metal grains is too large, an increase in the ratio of the conductive components in the wiring resistor 10 may decrease the specific resistance. On the other hand, if the amount of noble metal grains is too small, although the specific resistance increases in a preferable manner, the amount of glass frit relatively increases. Thus, the expansion is apt to occur. The wiring resistor 10 is also apt to expand when the amount of glass frit is too large. On the other hand, if the amount of glass frit is too small, the expansion rarely occurs but it becomes difficult to increase adhesion.

It is preferred that bismuth (Bi) or bismuth oxide (Bi_2O_3) be included in the noble metal paste P1. In other words, test results have shown that by adding these substances, expansion is suppressed even if the application amount of the added glass frit is large (10% or greater relative to the noble metal). Further, adhesion of the wiring resistor 10 is improved.

These substances are relatively easily oxidized and reduced in comparison to other oxides. It is presently presumed that such properties contribute to suppress expansion and enhance adhesion in one way or another.

When selecting, for example, aluminum nitride, as the substrate material, bismuth oxide reacts with the aluminum nitride when the paste is bonded and produces alumina and nitrogen gas. Thus, the bismuth oxide functions as an oxidization agent of the aluminum nitride. Further, when exposed to air, bismuth is easily oxidized into bismuth oxide.

Additionally, when selecting, for example, silicon nitride as the substrate material, bismuth oxide reacts with silicon nitride when the paste is bonded and produces silica and nitrogen gas. Thus, the bismuth oxide functions as an oxidization agent of the silicon nitride. In the same manner, bismuth may be considered as an oxidization agent of the silicon nitride.

It is preferred that about 1wt% to 10wt% of bismuth or bismuth oxide be included in the noble metal paste P1, more preferred that about 5wt% to 10wt% be included, and especially preferred that about 7wt% to 8wt% be included. If the content of bismuth or bismuth oxide is too small, the effect obtaining by adding bismuth or bismuth is insufficient. Thus, the expansion may not be prevented and the adhesion may not be significantly improved. On the other hand, if the content of bismuth and bismuth oxide is too large, the noble metal does not mix with the bismuth or the bismuth oxide and result in different resistances.

Further, the noble metal paste P1 must include ruthenium oxide (RuO_2). It is considered that together with bismuth or bismuth oxide, ruthenium oxide appropriately suppresses reaction between the glass frit and the aluminum nitride of the ceramic substrate and prevents the generation of reaction gas.

It is preferred that about 0.5wt% to 5wt% of ruthenium oxide be included in the noble metal paste P1, and particularly preferred that about 1wt% to 2wt% be included.

If the amount of ruthenium oxide is too small, the suppression of the reaction caused by bismuth or bismuth

oxide may not be guaranteed and the prevention of the expansion when the added amount of glass frit is too large may not be guaranteed. On the other hand, reaction caused by bismuth or oxide bismuth may be excessively suppressed when the amount of ruthenium oxide is too large, and the adhesion may not be improved when the added amount of glass frit is too large. It is preferred that the content of ruthenium oxide be less than or equal to the content of bismuth or bismuth oxide.

It is preferred that glass frit including zinc boron silicate (SiO_2 : B_2O_3 : ZnO_2) be used, and especially preferred that the glass frit includes zinc boron silicate as a main component. More specifically, it is preferred that a small amount of oxide be added to the zinc boron silicate. Specific examples of oxides include aluminum oxide (Al_2O_3), yttrium oxide (Y_2O_3), lead oxide (PbO), cadmium oxide (CdO), chromium oxide (Cr_2O_3), and copper oxide (CuO). One of these oxides or a combination of two or more of these oxides may be added to the zinc boron silicate. During the bonding of the paste, these oxides function as an oxidization agent of the substrate material and are thus reduced.

It is preferred that the weight ratio of each of the above listed oxides be 1/20 times to 1/5 times the weight ratio of zinc boron silicate, which serves as a base. If the weight ratio is too small, the percentage of the above oxides in the glass frit increases. As a result, the expansion caused by nitrogen gas may not be sufficiently prevented. On the other hand, if the weight ratio is too large, the percentage of the above oxides in the glass frit decreases. As a result, the adhesion of the wiring resistor may not be sufficiently increased.

The noble metal paste P1 also includes 2wt% to 15wt% of a resin binder, which serves as an organic vehicle, and 10wt% to 30wt% of a solvent. Examples of the resin binder are, for example, the cellulose group such as ethyl cellulose. The solvent is a component added to improve the printing and dispersion characteristics. Specific examples of the solvent are the acetate group, the cellosolve group such as butyl cellosolve, or the Carbitol group such as butyl Carbitol. One or a combination of two or more of these solvents may be used.

When the noble metal paste P1 applied to the ceramic substrate 9 is heated for a predetermined time at a temperature of about 750°C, the solvent in the noble metal paste P1 volatilizes and bonds the wiring resistor 10 and the pads 10a to the ceramic substrate 9. Fused glass frit has a tendency to move toward the ceramic substrate 9. Contrarily, the noble metal grains have a tendency to move away from the ceramic substrate 9.

Subsequently, the pads 10a are connected to the terminal pins by a solder S1 to complete the hot plate 3. Then, the hot plate 3 is attached to the opening 4 of the casing 2 to complete the desired hot plate unit 1 of Fig. 1.

(Examples and Comparative Examples)

[Preparation of Samples 1 to 8]

In examples 1 to 5 and comparative examples 1 to 3, 4 parts by weight of Y_2O_3 (average grain diameter $0.4\mu m$) and 8 parts by weight of an acrylic resin binder (manufactured by

Mitsui Chemicals, product name: SA-545, acid number 1.0) were added to 100 parts by weight of aluminum nitride powder (average grain diameter $1.1\mu\text{m}$) and mixed. The mixture produced in this manner was uniformly kneaded. The kneaded product was put into a press mold and pressed to form a plate-like molded product.

Then, after forming holes and performing a drying process, the molded product was degreased for four hours at a temperature of 350°C for four hours to thermally decompose the binder. Further, the degreased molded body was baked in a hot-press for three hours at a temperature of 1600°C to produce the aluminum nitride substrate, or the ceramic substrate 9. The pressure of the hot press was $150\text{kg}/\text{cm}^2$.

Then, after cutting the substrate and performing surface grinding, a paste applying process was performed. In the process, the noble metal paste P1, the composition of which is described below, was used and applied to a thickness of about $25\mu\text{m}$. Eight types of samples were prepared in accordance with the above procedure (refer to table 1).

Only one type of noble metal grains, that is, silver grains, which were flake-like and had an average grain diameter of $5\mu\text{m}$, was used. The added amount of the silver grains in the silver paste, which served as the noble metal paste P1, was 45wt% in sample 5, 50wt% in samples 2, 4, and 7, 55wt% in samples 1, 3, and 6, and 70wt% in sample 8.

The glass frit included zinc boron silicate as a base (i.e., a zinc-containing material was used as the glass frit). The amount of glass frit added in each sample is

shown in table 1, and the specific composition of each sample is shown in the lower rows of table 1. The amount of bismuth and ruthenium oxide that was added in each sample is as shown in table 1.

5

Ethyl cellulose was selected as the binder, and butyl Carbitol was selected as the solvent.

10 Although bismuth was added, ruthenium oxide was not added in samples 6, 7, and 8. Thus, samples 6, 7, 8 do not satisfy the optimal conditions of the present embodiment. Further, in sample 8, the amount of glass frit is small in comparison to the amount of silver grains. Thus, sample 8
15 does not satisfy the optimal conditions of the present embodiment. Accordingly, samples 1 to 5 correspond to examples 1 to 5, and samples 6 to 8 correspond to comparative examples 1 to 3.

[Comparison Test and Results]

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In each of the eight samples, the paste was printed to and bonded on the ceramic substrate 9, and two square millimeter test patterns were formed at multiple locations. The expansion of the test patterns was confirmed through
25 observation with the naked eye and with an optical microscope. A tensile strength test was performed on test patterns that did not expand, and the average value of the measured values (kgf/2mm \square) was calculated. Simultaneously, the resistance was measured with a Multimeter, and the
30 specific resistance ($\mu\Omega\cdot\text{cm}$) of the patterns was calculated from the measured length and the cross-sectional area of the patterns. The target value of the specific resistance was 10 $\mu\Omega\cdot\text{cm}$. The results of the experiments are shown in table 1.

In example 6, 20 parts by weight of Y_2O_3 (average grain diameter $0.4\mu\text{m}$), 15 parts by weight of Al_2O_3 (average grain diameter $0.5\mu\text{m}$), 20 parts by weight of SiO_2 (average grain diameter $0.5\mu\text{m}$), and 8 parts by weight of an acrylic resin binder (manufactured by Mitsui Chemicals, product name: SA-545, acid number 1.0) were mixed with 45 parts by weight of silicon nitride powder (average grain diameter $1.1\mu\text{m}$).

Then, after forming holes and performing a drying process, the molded product was degreased for four hours at a temperature of 350°C for four hours in a nitrogen atmosphere to thermally decompose the binder. Further, the degreased molded body was baked in a hot-press for three hours at a temperature of 1600°C to produce a silicon nitride substrate, or the ceramic substrate 9. The pressure of the hot press was 150kg/cm².

Then, after cutting the substrate and performing surface grinding, a paste applying process was performed. In the process, the noble metal paste P1, the composition of which is described below, was used and applied to a thickness of about 25 μ m. Bismuth oxide was used instead of bismuth.

Noble metal grains: 100 parts by weight of palladium grains (Pd-730 manufactured by Shoei Chemical Inc.)

Glass Frit: 2.0 parts by weight of SiO_2 , 5.0 parts by weight of B_2O_3 , 10.0 parts by weight of ZnO , and 1.2 parts by weight of PbO

Bi_2O_3 : 1.1 parts by weight

RuO_2 : 1.0 parts by weight

Resin binder: 3.4 parts by weight

Butyl Carbitol serving as a solvent: 17.9 parts by weight

The applied noble metal paste P1 was heated at a temperature of about 750°C for a predetermined time to bond the wiring resistor 10 and the pads 10a and complete sample 9, which corresponds to the hot plate 3 of example 6.

[Comparison Test and Results]

The same comparison test as that conducted on examples 1 to 5 and comparison examples 1 to 3 was performed on sample 9. Expansion of the wiring resistor 10 was not

confirmed. Further, the tensile strength test was 11.5kgf/2mm² and greater than examples 1 to 5. The specific resistance of the patterns was 110 $\mu\Omega$ ·cm and much greater than the target value.

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Accordingly, the examples of the present embodiment have the advantages described below.

10 (1) In the hot plates 3 of examples 1 to 6, the added amount of glass frit is adjusted at 10wt% or greater relative to the noble metal grains to increase the specific resistance of the conductive pattern layer (i.e., the wiring resistor 1 and the pads 10a).

15 Accordingly, the ratio of the conductive components in the wiring resistor 10 is small and the specific resistance is extremely large. Thus, the hot plate 3 has a superior heating capability. Further, such hot plate 3 is optimal when used, for example, for heating at high temperatures
20 (200°C or greater).

(2) The wiring resistor 10 and the pads 10a of the hot plate 3 in examples 1 to 5 are formed from ruthenium oxide, bismuth, glass frit, and silver grains. Further, the wiring
25 resistor 10 and the pads 10a of the hot plate 3 in example 6 are formed from ruthenium oxide, bismuth oxide, glass frit, and palladium grains.

Accordingly, the synergism of bismuth or its oxide and
30 ruthenium oxide prevents the expansion of the wiring resistor 10 even if the added amount of glass frit is relatively large to increase the specific resistance. Therefore, the wiring resistor 10 does not expand and has a

In examples 1 to 5, the bismuth in the noble metal paste P1 may be replaced by the same amount of bismuth oxide. In example 6, the bismuth oxide in the noble metal paste P1 may be replaced by the same amount of bismuth.

(3) In examples 1 to 5, an aluminum nitride substrate, which has especially superior heat resistance and high thermal conductivity, is used as the ceramic substrate 9. Thus, the hot plate 3 is practical since it may be used under high temperatures.

(4) The noble metal paste P1 used in examples 1 to 6 includes 1wt% to 10wt% of bismuth or bismuth oxide, which is an optimal amount. Thus, expansion is prevented, adhesion is improved, and the specific resistance is increased.

(5) The noble metal paste P1 used in examples 1 to 6 includes 0.5wt% to 5wt% of ruthenium oxide, which is an optimal amount. Thus, expansion is prevented, adhesion is improved, and the specific resistance is increased.

The embodiment of the present invention may be modified as described below.

Spherical noble metal grains may be used in lieu of the flake-like noble metal grains. Further, instead of using only one type of the noble metal grains, two or more types

The oxide included in the glass frit, the base of which is zinc boron silicate, is not limited to Al_2O_3 , which is used in the examples of the above embodiment, and may, of course, be changed.

The ceramic substrate 9 is not limited to products manufactured through a press molding and may be manufactured, for example, by performing sheet molding with a doctor blade apparatus. When performing sheet molding, the wiring resistor 10 may, for example, be arranged between superimposed sheets. Thus, the high temperature hot plate 3 is manufactured in a relatively simple manner.

The conductive pattern layer is not limited to the wiring resistor 10 and the pads 10a used in the above embodiment and may be other structures.

The noble metal paste P1 need not be screen printed on the ceramic substrate 9. For example, the noble metal paste P1 may be stamped on the ceramic substrate 9.

Article 34 Amendment

1. A hot plate that uses a ceramic substrate provided with a conductive layer, the hot plate characterized in that the conductive layer includes ruthenium oxide, glass frit, and noble metal grains.
2. The hot plate according to claim 1, characterized in that the conductive layer includes ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains.
3. The hot plate according to claim 1 or 2, characterized in that the ceramic substrate us a ceramic nitride substrate or a ceramic carbide base plate.
4. The hot plate according to any one of claims 1 to 3, wherein the glass frit includes zinc boron silicate.
5. The hot plate according to any one of claims 1 to 4, wherein the noble metal grains is at least one selected from gold grains, silver grains, platinum grains, and palladium grains.
6. (Amended) A wiring resistor conductive paste used for a ceramic hot plate characterized in that the

wiring resistor conductive paste includes ruthenium oxide, glass frit, and noble metal grains.

7. (Amended) A wiring resistor conductive paste used in a ceramic hot plate characterized in that the wiring resistor conductive paste includes ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains.

ABSTRACT

5 A hot plate that does not expand much, has superior adhesion, and is provided with a conductive pattern layer having a large specific resistance. The hot plate 3 uses a ceramic nitride substrate 9, which includes conductive patterns 10, 10a. The conductive pattern layers 10, 10a is formed from ruthenium oxide, bismuth or its oxide, glass frit, and noble metal grains.

Fig. 1

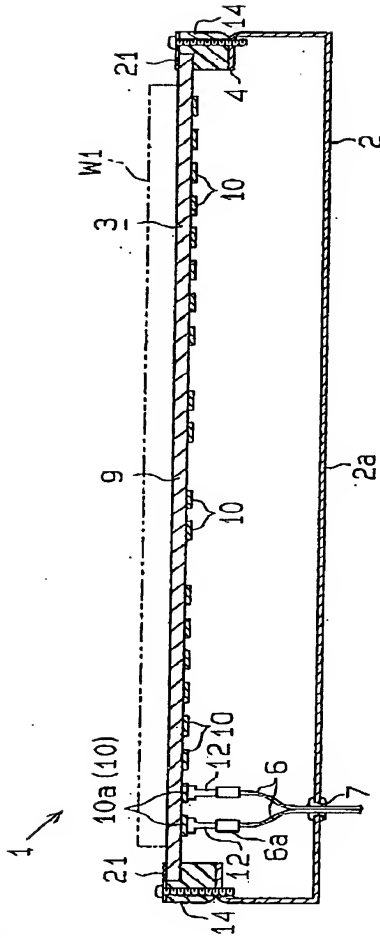
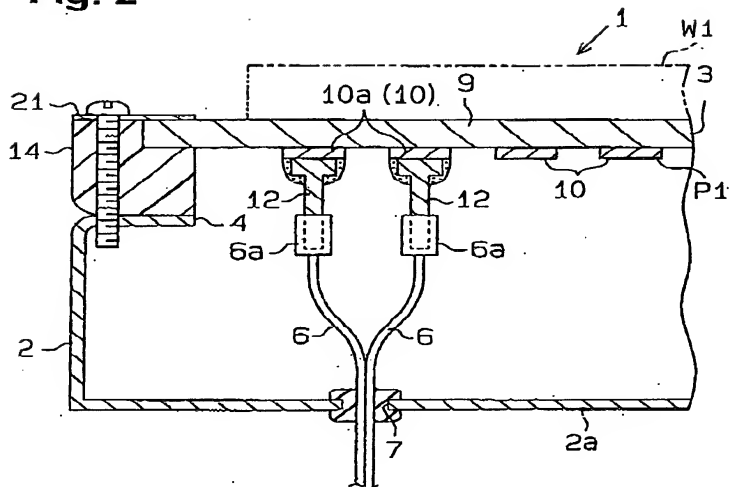


Fig. 2

COMBINED DECLARATION AND POWER OF ATTORNEY

Docket:

As a below named inventor, I hereby declare that:

My residence post office address and citizenship are as stated below next to my name.

I believe I am the original first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled **HOT PLATE AND CONDUCTIVE PASTE**
 the specification of which

(check one)

☐
☒
is attached hereto May 1, 2000

was filed on

as US Application Serial Number or PCT International Application Number

PCT / JP00 / 02874and was amended on October 6, 2000 (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability as defined in 37 CFR § 1.55.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Not Claimed

Prior Foreign Application(s)

Pat. Appln. No. 11-126973 Japan07 / 05 / 1999

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Pat. Appln. No. 11-323061 Japan(Day/Month/Year Filed)12 / 11 / 1999

[]

(Day/Month/Year Filed)**SEE ATTACHED SUPPLEMENTAL SHEET**

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

(Application Number)

(Filing Date)

(Application Number)

(Filing Date)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365 of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

(Application Number)

(Filing Date)

(Status: patented, pending, abandoned)

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(Filing Date)

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I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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SUPPLEMENTAL SHEET

ADDITIONAL PRIOR FOREIGN APPLICATION(S)

Number	Country	Day/Month/Year	Priority Benefits Claimed?
Pat. Appln. No. 2000-126786	Japan	27 / 04 / 2000	Yes